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PROGRESS REPORT ON QUARTZ ANALYSIS IN MINE DUST BY INFRARED ABSORPTION

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March 1982

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Project MRP-3.2.0.0.01
Respirable Dust

39 pp

MINERALS RESEARCH PROGRAM
MINERAL SCIENCES LABORATORIES
DIVISION REPORT MRP/MSL 82-40(TR)

MICROMEDIA
OCT 27 1982

PROGRESS REPORT ON QUARTZ ANALYSIS IN MINE DUST BY INFRARED ABSORPTION

by

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SUMMARY

Development of an infrared absorption method for the determination of a quartz dust deposited on filter membranes has continued. Calibration curves were established using quartz deposits produced using a CAMPEDS air sampler. The method was checked by ashing filter membranes with quartz samples, depositing these onto other filters from a liquid suspension, and redetermining the quartz using calibration curves established independently. Non-uniformity of filter membranes and changes in the infrared absorption of recently manufactured membranes are sources of difficulty for the method. A standard has been developed for verifying long-term instrumental stability. Investigations are continuing to improve the method and to establish its applicability to dust samples containing other components in addition to quartz.

INTRODUCTION

A study to assess the feasibility of determining α-quartz quantitatively in mine dust, directly on the filter membrane upon which it is deposited during air sampling, using infrared spectroscopy, is under continuing investigation as part of the Energy, Mines and Resources Canada research under CANMET project No. 3.2.0.0.01 "Respirable Dust". The report documents the progress made since March 1981.

EXPERIMENTAL STUDIES AND RESULTS

CALIBRATION CURVES BASED ON AIR DEPOSITED QUARTZ STANDARD SAMPLES

Quartz deposits prepared by air deposition are inhomogeneous across their area and quantitative determination cannot be made using calibration curves obtained using liquid deposited standards that are homogeneous (1). Pure quartz standard samples prepared at the Elliot Lake Mining Research Laboratories were collected on a CAMPEDS sampler in a dust chamber (2) in order to reproduce the particle size distribution and the particle deposition pattern on the area of the filter that samples taken in the field would have. The weight of quartz deposited was determined gravimetrically. The area of the deposit is 3.1416 cm^2.

The standard samples, deposited on Gelman DM5000 filters, were read on a Beckman model 4260 double beam spectrophotometer with a portion cut from the same filter in the reference beam using the following conditions: wave number span 950-680 cm^{-1}, a fixed slit of 4 mm, scan speed of 300 cm^{-1}/min, a period of 1 sec and a gain of 0.024. The absorbance of each sample was read in four positions equidistant around the circumference of the circular deposit. A sample showing large differences in these four readings was not used for the determination of the calibration curve.

Forty-five samples were used. Area of the quartz doublet and the heights of the 800 cm^{-1} and 780 cm^{-1} peaks were determined and plotted against the weight of quartz on the filter (Fig. 1, 2, 3 respectively). A scale for the mean surface concentration (weight/unit area) of quartz on the filters is also shown in these figures. Equations for linear regression, linear regression through the origin, parabolic regression, parabolic regression through the origin, hyperbolic regression, hyperbolic regression through

the origin and their correlation coefficients were calculated (Tables 1, 2, 3 respectively). The parabolic regressions gave the best correlation coefficient and were plotted (Fig. 1, 2, 3 respectively).

Because of the uncertainty of the low weight, the calibration range was extended much beyond the area of interest. A proposed threshold limit value is 100 µg respirable quartz per cubic metre, approximately the volume sampled in an eight-hour shift. To check the validity of the parabolic regression at the lower end of the calibration curve, it was also calculated using only readings of samples of 420 µg quartz and less and only readings of samples of 220 µg quartz and less (Tables 4, 5) (Fig. 4). Although the regression equations obtained were different, they gave an acceptably similar calculated value for the weight of quartz (Table 6).

The weight of quartz was determined, using the parabolic regression equations given in Tables 1 and 2 for a set of air desposited standard samples prepared in 1978. These values are compared with gravimetric values obtained using X-ray diffraction (XRD) (Table 7a). The mean weight of quartz and the standard deviation calculated from IR peak area measurements were plotted against the gravimetric values (Fig. 5, 6, 7).

Plots such as those in Fig. 5 and 6 ideally should show straight lines passing through the origin with slopes equal to 1 for perfect agreement between methods. The values of the slopes and intercepts and of the correlation coefficients calculated by regression analysis give an indication of the agreement between pairs of methods.

The results of such analysis are shown in Table 7b for selected pairs of methods. It appears from the parameters in this table that both infrared absorption and X-ray diffraction, compared to the gravimetric determination, have overestimated the mass of quartz in the deposits at lower values (cf, Y intercepts) and underestimated the mass for higher values (cf slopes). The correlation coefficient between the gravimetric data and the X-ray diffraction data is higher than the correlation coefficients with the IR determinations. The IR results, however, may have been influenced by loss of deposit in handling; this could account for the anomalously low result for sample 12 in Table 7a. Also, for comparison with the calibration curves, regression equations were calculated using the peak area values obtained for this set of samples (Table 8) and a plot of parabolic regression superimposed on the corresponding calibration curve is shown in Fig. 8 and 9. The slope at the lower end of the

parabolic regression is smaller and the intercept at zero weight is larger for the earlier set of samples.

LOW TEMPERATURE ASHING AND REDEPOSITION OF QUARTZ DEPOSITS

In order to increase sensitivity and decrease the problem of inhomogeneous thickness of air deposits and matrix interference in methods using IR to determine quartz in dust, the collecting membrane and dust deposit are often ashed, and the ashed material is either redeposited from a liquid suspension onto another membrane using a smaller deposit area or incorporated into a KBr pellet (3,4). A method for low temperature ashing and liquid redeposition was investigated in order to obtain comparison values for air deposited samples.

To avoid some of the problems associated with ashing at higher temperatures, such as sample loss and distortion of the silica doublet, samples were ashed at <100°C in a low temperature plasma asher. A fine white residue remained. This residue adherred to the beaker on which it was ashed and could not be dispersed ultrasonically in isopropanol as can residues ashed at 650°C (3). Water is unsuitable for the suspension of quartz unless a wetting agent is used because droplets adhere to the funnel used for redeposition. Initial treatment with water followed by the addition of isopropanol to the slurry allowed transfer of the residue. The absorption peaks exhibited by the ash residue from a filter membrane without quartz (Fig. 10), probably representing frequencies of a tetrahedral XY_4 group, did not interfere with the quartz doublet. The spectrum of a membrane ashed at 650°C is shown for comparison (Fig. 11).

A series of quartz deposits was prepared on DM450 membranes by liquid deposition using a procedure previously described (1) and examined by IR absorption. A half of each membrane was used as a reference. Considerable variation was found between the two halves of some DM450 filters currently produced, so examination before deposition was necessary. Each filter membrane with its deposit was placed, deposit side down, in a 50 mL beaker and ashed for two hours at 100 watts and three hourse at 250 watts with an oxygen flowrate of 300 mL/min. The ashed deposit and the corresponding ashed reference portion of the filter were redeposited on separate halves of another DM450 filter after each had been mixed with 2 mL of water and, upon the addition of 8 mL of isopropanol, slurried for fifteen minutes. The weights of

quartz on the filter before and after redeposition (Table 9) were calculated from a parabolic regression equation based on peak areas obtained for the quartz doublet using standard deposits of quartz of the same particle size, prepared as described previously (1).

Four deposits from the 1978 set of air deposited samples from Elliot Lake were ashed and redeposited using the above method except that there was no corresponding ashed blank filter for redeposition. However the DM5000 filter used in air deposition, which has a pore size greater than ten times that of the one used for liquid deposition, produces much less residue. The weight of quartz was calculated using a regression equation prepared from data on sample deposits of quartz of a similar particle size (Table 7). Regression analysis on the limited results available indicated excellent correlation between the masses of quartz determined before and after redeposition (Table 7b). The intercept was negligable, but the value of the slope indicated that the mass determined after redeposition tended to be low.

FILTER MEMBRANES

Gelman Metricel DM membranes composed of a vinyl chloride-acrylonitrile copolymer were chosen as the collection filter for the project because quartz can be determined directly on them by both IR and XRD, allowing the same sample to be analyzed by both techniques. DM5000, having a pore size of 5 µm, is used for air deposition while DM450, with a pore size of 0.45 µm, is used for liquid deposition. Polyvinyl chloride membranes are suitable for use in the direct IR determination but not for the determination using XRD (5). In the past DM5000 membranes have exhibited relatively uniform absorbance in the region of the quartz doublet (Fig. 12a). This absorbance was compensated for by placing a portion cut from the same filter in the reference beam. A horizontal slightly wavy baseline usually resulted. In actual samples collected in mines using personal dust samplers the reference portion of the same filter will not be available. A series of filters from different lots currently produced by Gelman were examined to see how uniform the filters were within the same box, within the same lot, and within different lots. Examination of the spectra of currently produced filters showed greater absorption and greater slope in the region of the quartz doublet (Fig. 12b).

Ten filters were taken at random from each of two boxes from the first lot and from one box from the second, third and fourth lots. The variation in the weight of quartz calculated from the areas obtained for the quartz doublet when one selected deposit was read with different filters in the reference beam showed the effect of non-uniformity of the filters. A histogram and analysis of variance of values for the weight of quartz obtained using filters from two boxes in one lot is shown in Fig. 13, and using boxes of different lots in Fig. 14. The calculated weight of quartz can vary as much as from 129 μg to 107 μg when using different filters from the same box in the reference beam. Analysis of variance gives an F ratio of 7.12, with $F_{.95}$ (1,18) = 4.41, using filters from two boxes in the same lot, and an F ratio of 9.93 with $F_{.95}$ (3,46) = 2.81, using filters from different lots. It is concluded, therefore, that there can be significant differences between filters from different lots and from different boxes of the same lot.

It has been suggested that if variation in the absorption of filters is due to differences in filter thickness, computer processing of data could eliminate background spectra by equalization using the size of a chosen band in the filter spectrum. Some success was reported using polyvinylchloride filters (6). In order to examine the possibilities of this technique using DM filters, the lower end of the wave number range examined in the filter study was extended from 680 cm^{-1} to 540 cm^{-1} to include the 630 cm^{-1} band of the DM spectrum. The area of this peak, which was determined along with the area of the quartz doublet, reflected the incomplete compensation of the filter in the sample beam by the filter in the reference beam due to differences in filter thickness. The DM filter spectrum has the low frequency end of a strong absorption band in the region of the quartz doublet (Fig. 12), and this would affect the area obtained for the doublet if not compensated for exactly by the filter in the reference beam. A plot of the area of the quartz doublet against the area of the 630 cm^{-1} peak of the DM filter (Fig. 15) shows a rough trend to larger areas for the quartz doublet when the area of the 630 cm^{-1} peak decreases, but the correlation between the two areas is not good enough to provide a reliable basis for adjusting the measured areas of the quartz doublet. Even when no difference was detected in the 630 cm^{-1} peak of the two filters, slight differences in the large peak on the high frequency side of the region of the quartz doublet affected the position of the first

minimum, i.e., the high frequency limit of the quartz doublet, and consequently the value calculated for the weight of quartz.

Autoscaling of the DM spectrum based on measurement of the peak height of the 630 cm^{-1} peak from the zero baseline was not attempted at this time because of instrumental problems with exact resetting of the initial wavenumber of the region of the spectrum measured, a problem that is not critical in the computation of the peak height and peak area using the baseline method. A further complication in the use of the 630 cm^{-1} peak for thickness compensation could result from the fact that cristobalite, a polymorph of silica which can be present in mine samples, exhibits an intense absorption band at 620 cm^{-1}. Latest communications with the Gelman Instrument Co. reveal that they are now discontinuing production of DM5000 until solutions can be found for their manufacturing problems. In the interim, for this project mine dust deposits are being prepared on filters that have been cut into two parts from larger filters to allow for compensation using the other part of the filter in the reference beam of the spectrophotometer during determination of the quartz.

PERMANENT STANDARDS

A permanent standard was required to monitor day to day instrument performance. A dispersion of quartz throughout a solid medium could not be prepared because any matrix material, although stable and inert to general laboratory conditions and having no significant absorption peaks in the region of the quartz doublet, had sufficient background absorption to prevent using anything but a very thin film. Standards prepared for calibration use by deposition of quartz powder on filters were obviously not reliable for long term use, but several methods have been tried to make them more permanent. A second filter was placed over the surface of the deposit and the blank edges sealed with heat. The covering filter could not be sealed across the deposit because of severe wrinkling. In another approach a DM filter containing a quartz deposit was dissolved in acetone and allowed to resolidify. In order to produce the same thickness of DM material, the filter was held in a mask while several drops of acetone were placed on the membrane behind the deposit, dissolving it, but keeping it as a liquid film. Upon evaporation of the acetone the quartz was embedded in or stuck to the resolidified membrane. Standards prepared by the above methods along with similarly treated blanks to be

used in the reference beam are being monitored to determine their long term stability.

While any standard material whose state and concentration is absolutely reproducible or constant could be used for checking frequencies and absorption intensities, the best would be one which would provide a peak of acceptable size and position in the IR spectrum so that its area and peak height could be determined using the same conditions and procedure as those used for the quartz doublet when samples were analyzed. Materials such as nitrates and carbonates exhibit a stretching frequency in the IR spectrum in the region of the quartz doublet (ν_2 of planar XY_3 groups). Some collection filters are composed of these materials and several were examined. A polycarbonate filter made by Nuclepore Corp. is of such a thickness that it exhibits an absorption peak of an acceptably measurable size. One of these filters has been permanently mounted and the peak area measurement can be obtained using the same program as that used for the quartz doublet with a slight modification. Data is collected in the spectral region of 950 cm^{-1} to 680 cm^{-1} but only the 800-700 cm^{-1} portion is examined for detection of minima and integration of the peak. This standard is used at the present time to monitor instrument performance (Fig. 16).

BACKGROUND MATRIX

The effects of background matrix from minerals and organics in mine dust and of cristobalite, a polymorph of quartz present in some mine and mineral processing dusts, are being investigated.

CONCLUSIONS AND RECOMMENDATIONS

The development of an on-filter method for the infrared determination of quartz has progressed. Calibration curves have been established for pure quartz dust samples as collected on DM5000 membranes by the CAMPEDS air sampler. Although distinctly different calibration curves were obtained for quartz deposited from liquid suspension onto DM450 membranes, the calibrations for the two types of deposits were successfully interrelated by ashing samples originally deposited on DM5000 membranes and redepositing them onto DM450 membranes from liquid suspension. Since the establishment of a reliable calibration is critical, some further verification of the calibration curves will be made.

Difficulties have been encountered with non-uniformity and fairly high IR absorption by DM5000 filter membranes of recent manufacture. It will be necessary to select the best commercially available filter membrane for quartz determination, which may not be the current DM5000 filters.

Two standards for monitoring instrument stability have been developed; the better one will be selected and used for routine application.

The applicability of the infrared method for the determination of quartz in the presence of other components must be established. Investigations should include examination of actual field samples and the study of infrared spectra of other minerals likely to be found in mine dusts.

ACKNOWLEDGEMENTS

The author wishes to thank M. Curtin, a student assistant, who provided the IR data for the air deposited quartz samples and the filter study, G. Knight, Elliot Lake Mining Research Laboratories, who supplied the air deposited samples and the results of gravimetric determinations of the deposit weights, R.F. Pilgrim, Physical Chemistry Section, for the development and modification of the computer programs and J.H.G. Laflamme, Mineralogy Section, Mineral Processing Laboratory for low temperature ashing of the deposits.

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CALIBRATION CURVE ELLIOT LAKE AIR DEPOSITS AREA

LINEAR REGRESSION THROUGH ZERO - Y = B * X

B = 1.65427E+01 CORR.COEF = 9.97357E-01

LINEAR REGRESSION - Y = A + B * X

A = 4.70134E+02 B = 1.61839E+01 CORR.COEF = 9.97951E-01

PARABOLIC REGRESSION THROUGH ZERO - Y = B * X + C * X * X

B = 1.86287E+01 C = -1.40540E-03 CORR.COEF = 9.97794E-01

PARABOLIC REGRESSION - Y = A + B * X + C * X * X

A = 3.55858E+02 B = 1.73277E+01 C = -7.11859E-04 CORR.COEF = 9.98028E-01

HYPERBOLIC REGRESSION THROUGH ZERO - Y = B*X/(1+C*X)

B = 1.74730E+01 C = 3.80378E-05 CORR.COEF = 9.97523E-01

HYPERBOLIC REGRESSION - Y = (A+B*X)/(1+C*X)

A = -5.16493E+01 B = 1.60818E+01 C = -2.04548E-05 CORR.COEF = 9.97226E-01

Table 1. Regression equations calculated using area of the quartz doublet.
x = weight of quartz on the filter (µg)
y = peak area (absorbance x 10^3 x cm^{-1})

CALIBRATION CURVE ELLIOT LAKE AIR DEPOSITS PK.HT(800)

LINEAR REGRESSION THROUGH ZERO - Y = B * X

B = 4.98456E-01 CORR.COEF = 9.97907E-01

LINEAR REGRESSION - Y = A + B * X

A = 4.03111E+00 B = 4.95380E-01 CORR.COEF = 9.97954E-01

PARABOLIC REGRESSION THROUGH ZERO - Y = B * X + C * X * X

B = 5.30790E-01 C = -2.17839E-05 CORR.COEF = 9.98019E-01

PARABOLIC REGRESSION - Y = A + B * X + C * X * X

A = 7.77271E-01 B = 5.27948E-01 C = -2.02691E-05 CORR.COEF = 9.98020E-01

HYPERBOLIC REGRESSION THROUGH ZERO - Y = B*X/(1+C*X)

B = 4.94776E-01 C = -4.98482E-06 CORR.COEF = 9.97928E-01

HYPERBOLIC REGRESSION - Y = (A+B*X)/(1+C*X)

A = -8.32582E-03 B = 4.94558E-01 C = -5.28810E-06 CORR.COEF = 9.97928E-01

Table 2. Regression equations calculated using the height of the 800 cm^{-1} band.
x = weight of quartz on the filter (µg)
y = peak height (absorbance x 10^3)

CALIBRATION CURVE ELLIOT LAKE AIR DEPOSITS PK.HT(780)

LINEAR REGRESSION THROUGH ZERO - Y = B * X

B = 4.21314E-01 CORR.COEF = 9.97782E-01

LINEAR REGRESSION - Y = A + B * X

A = 7.42916E+00 B = 4.15644E-01 CORR.COEF = 9.98007E-01

PARABOLIC REGRESSION THROUGH ZERO - Y = B * X + C * X * X

B = 4.68082E-01 C = -3.15093E-05 CORR.COEF = 9.98115E-01

PARABOLIC REGRESSION - Y = A + B * X + C * X * X

A = 3.45043E+00 B = 4.55467E-01 C = -2.47846E-05 CORR.COEF = 9.98148E-01

HYPERBOLIC REGRESSION THROUGH ZERO - Y = B*X/(1+C*X)

B = 4.40195E-01 C = 3.02971E-05 CORR.COEF = 9.97870E-01

HYPERBOLIC REGRESSION - Y = (A+B*X)/(1+C*X)

A = -9.30400E-01 B = 4.14159E-01 C = -1.26103E-05 CORR.COEF = 9.97730E-01

Table 3. Regression equations calculated using the height of the 780 cm^{-1} band.
x = weight of quartz on the filter (µg)
y = peak height (absorbance x 10^3)

AREA

LINEAR REGRESSION THROUGH ZERO - Y = B * X

B = 1.89629E+01 CORR.COEF = 9.92669E-01

LINEAR REGRESSION - Y = A + B * X

A = 2.84148E+02 B = 1.79123E+01 CORR.COEF = 9.96464E-01

PARABOLIC REGRESSION THROUGH ZERO - Y = B * X + C * X * X

B = 2.15442E+01 C = -7.32354E-03 CORR.COEF = 9.94435E-01

PARABOLIC REGRESSION - Y = A + B * X + C * X * X

A = 2.88480E+02 B = 1.78130E+01 C = 2.36251E-04 CORR.COEF = 9.96465E-01

HYPERBOLIC REGRESSION THROUGH ZERO - Y = B*X/(1+C*X)

B = 2.11927E+01 C = 3.37893E-04 CORR.COEF = 9.94293E-01

HYPERBOLIC REGRESSION - Y = (A+B*X)/(1+C*X)

A = -1.90820E+01 B = 1.86214E+01 C = -6.24480E-05 CORR.COEF = 9.92572E-01

Table 4. Regression equations calculated using area of the quartz doublet for standard samples $\leq$ 420 µg quartz.

x = weight of quartz on the filter (µg)

y = peak area (absorbance x 10^3 x cm^{-1})

AREA

LINEAR REGRESSION THROUGH ZERO - Y = B * X

B = 2.00158E+01 CORR.COEF = 9.75135E-01

LINEAR REGRESSION - Y = A + B * X

A = 2.87856E+02 B = 1.78600E+01 CORR.COEF = 9.88416E-01

PARABOLIC REGRESSION THROUGH ZERO - Y = B * X + C * X * X

B = 2.48056E+01 C = -2.75128E-02 CORR.COEF = 9.84099E-01

PARABOLIC REGRESSION - Y = A + B * X + C * X * X

A = 2.55760E+02 B = 1.89588E+01 C = -4.93091E-03 CORR.COEF = 9.88538E-01

HYPERBOLIC REGRESSION THROUGH ZERO - Y = B*X/(1+C*X)

B = 2.45341E+01 C = 1.33284E-03 CORR.COEF = 9.83249E-01

HYPERBOLIC REGRESSION - Y = (A+B*X)/(1+C*X)

A = -2.43752E+01 B = 1.94659E+01 C = -2.16045E-04 CORR.COEF = 9.76728E-01

Table 5. Regression equations calculated using area of the quartz doublet for standard samples $\leq$ 220 µg quartz.
x = weight of quartz on the filter (µg)
y = peak area (absorbance x 10^3 x cm^{-1})

Table 6 - Comparison of quartz values calculated using three different regression equations

Sample No.	Peak area (absorbance x cm^{-1})	Quartz a*	Quartz b*	Quartz c*
1	7.187	401	385	
2	3.962	210	206	207
3	2.072	99	100	98
4	1.401	60	62	61
5	.696	20	23	23
6	.413	3	7	8

* weight of quartz calculated from regression equations determined using standard sample weights:

$\leq$ 1710 µg (a)
$\leq$ 420 µg (b)
$\leq$ 220 µg (c)

Table 7a - Inter comparison of quartz methods

No.	Weight of quartz (µg)								
	Gravimetric	IR						XRD*	IR
		Peak area		800 cm^{-1} peak height		780 cm^{-1} peak height			following ashing and liquid redeposition
	weight	Mean†	Stand† dev.	Mean†	Stand† dev.	Mean†	Stand† dev.		
5	1733	1705	20	1667	21	1737	12	1685	
11	1006	932	18	896	15	920	22	859	
12	990	730	27	699	25	709	22	877	
13	980	917	28	857	21	901	25	891	
14	971	977	29	926	17	964	33	896	
15	981	970	21	920	16	962	20	876	
16	913	942	13	907	14	933	11	845	
17	691	624	21	611	19	614	21	655	
18	700	672	18	648	14	671	13	688	
20	647	686	6	654	5	678	5	705	
21	680	717	9	695	5	709	10	696	
22	689	764	21	740	13	761	17	721	706
23	684	690	16	689	13	687	18	680	
24	645	618	9	612	7	616	8	626	
25	210	206	5	198	6	205	6	266	
26	212	218	4	208	4	213	5	246	
27	225	236	5	230	2	238	3	258	
28	209	220	5	216	2	216	3	253	
29	207	193	2	190	2	193	3	234	
30	216	211	6	211	6	213	6	254	
31	214	213	4	209	5	217	5	247	
32	196	186	1	185	1	187	1	248	157
33	148	159	2	153	1	165	1	206	
34	152	162	5	156	2	161	3	200	
35	157	170	3	164	1	175	2	202	
36	154	182	1	163	1	176	2	194	
37	141	149	3	142	2	153	3	184	
38	154	170	3	157	2	169	2	206	
39	155	177	3	159	2	169	2	198	
40	135	146	2	138	1	146	1	162	136
53	35	50	2	53	1	55	1	64	53
54	42	58	4	58	2	61	2	69	
55	48	58	1	60	1	65	1	77	
56	37	72	1	59	1	70	1	70	
57	24	60	3	57	2	65	1	64	
58	44	51	3	57	2	61	3	75	
59	14	51	2	57	1	62	2	70	
60	18	33	1	39	1	43	1	56	

Table 7a (Cont'd)

No.	Weight of quartz (µg)							
	Gravimetric	IR					XRD*	IR
		Peak area		800 cm^{-1} peak height		780 cm^{-1} peak height		following ashing and liquid redeposition
	weight	Mean†	Stand† dev.	Mean†	Stand† dev.	Mean†	Stand† dev.	
62	120	124	2	127	1	131	1	149
63	122	130	4	131	4	140	5	156
64	115	125	4	118	4	132	5	150
65	129	130	3	128	2	133	4	149
66	117	138	3	129	2	137	2	162
67	131	133	2	125	2	140	2	144
68	117	138	5	134	4	137	5	160

* no correction for thick sample absorption or penetration of quartz into the membrane filter.

† values calculated on four readings

Table 7b - Comparison of methods of quartz determination by linear regression analysis

Method A (x)	Method B (y)	Slope	Interception y axis (µg)	Correlation coefficient	Number of samples
Gravimetric	IR (peak area)	0.945	19	0.993	45
Gravitmetric	IR (800 cm^{-1} peak ht.)	0.912	19	0.993	45
Gravitmetric	IR (780 cm^{-1} peak ht.)	0.940	22	0.992	45
Gravitmetric	XRD	0.895	51	0.996	45
IR (peak area)	IR (peak area after ashing and redeposition)	0.923	-2	0.999	4

Table 8 - Regression equations calculated from peak area values of 1978 Elliot Lake air deposits
x = weight of quartz on the filter (µg),
y = peak area (absorbance x 10^3 x cm^{-1})

LINEAR REGRESSION THROUGH ZERO - Y = B * X

B = 1.64818E+01 CORR.COEF = 9.87994E-01

LINEAR REGRESSION - Y = A + B * X

A = 8.22162E+02 B = 1.54285E+01 CORR.COEF = 9.92792E-01

PARABOLIC REGRESSION THROUGH ZERO - Y = B * X + C * X * X

B = 1.82215E+01 C = -1.68267E-03 CORR.COEF = 9.90022E-01

PARABOLIC REGRESSION - Y = A + B * X + C * X * X

A = 7.73661E+02 B = 1.57616E+01 C = -2.62077E-04 CORR.COEF = 9.92824E-01

HYPERBOLIC REGRESSION THROUGH ZERO - Y = B*X/(1+C*X)

B = 1.76339E+01 C = 6.88076E-05 CORR.COEF = 9.89777E-01

HYPERBOLIC REGRESSION - Y = (A+B*X)/(1+C*X)

A = -2.46118E+01 B = 1.63363E+01 C = -1.05750E-05 CORR.COEF = 9.88914E-01

Table 8. Regression equations calculated from peak area values of 1978 Elliot Lake air deposits.
x = weight of quartz on the filter (µg)
y = peak area (absorbance x 10^3 x cm^{-1})

Table 9 - Recovery of liquid deposited quartz after low temperature ashing and liquid redeposition**

No.	Initial		Final		Recovery
	Peak area* (absorbance x cm^{-1})	Quartz (µg)	Peak area* (absorbance x cm^{-1})	Quartz (µg)	%
1	17.041	615	17.035	615	100.0
2	6.961	244	6.843	239	98.3
3	3.549	120	3.505	118	98.7
4	1.925	62	1.899	60	98.6
5	.962	26	.943	25	98.0
6	.437	7.3	.420	6.4	96.1

* average of three readings

** the area of the quartz deposit is 1.416 cm^2

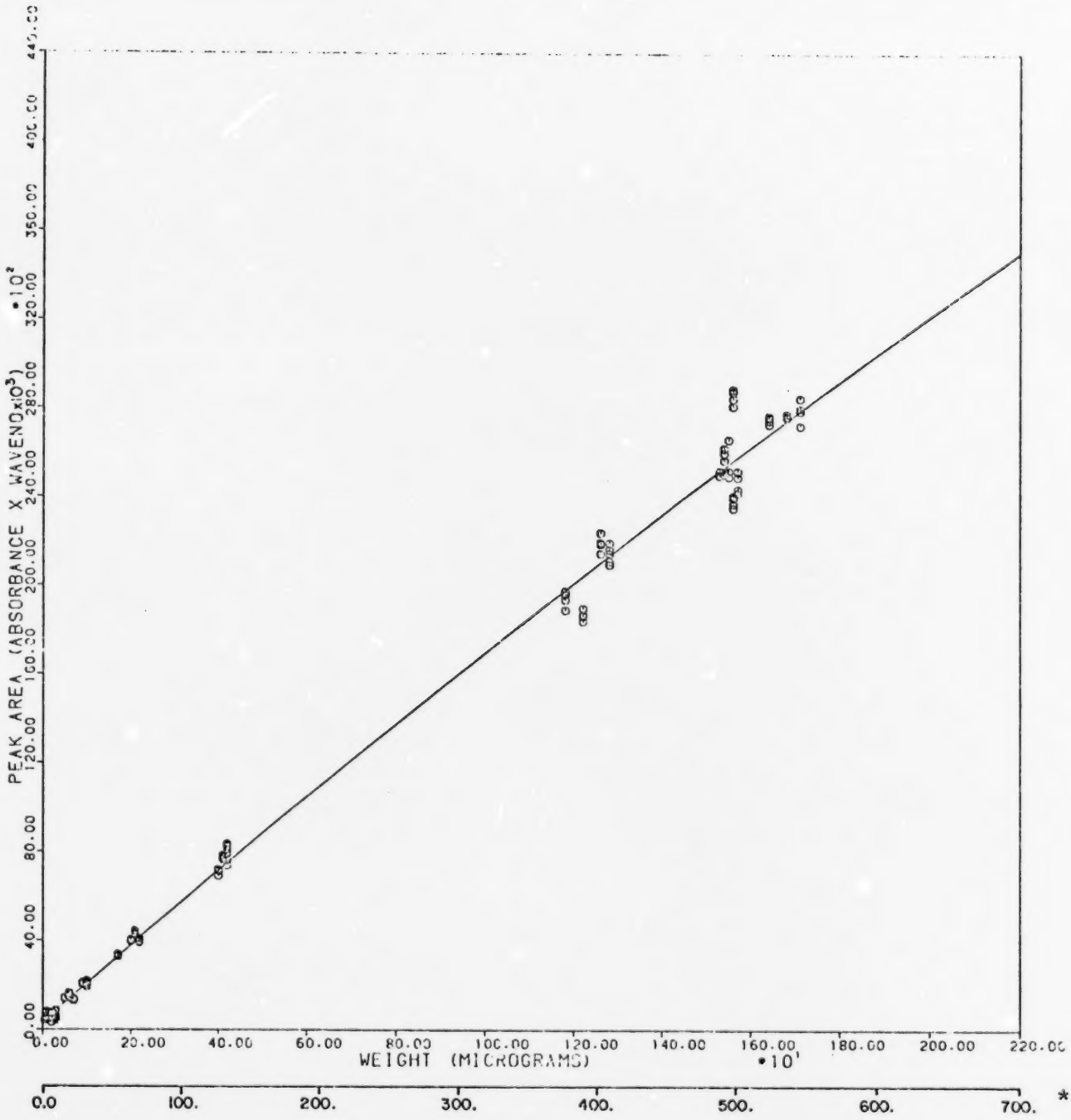


Figure 1. AIR DEPOSITED QUARTZ CALIBRATION CURVE
Plot of peak area values and the calculated parabolic regression line.
*surface density of the quartz deposit ($\mu g/cm^2$)

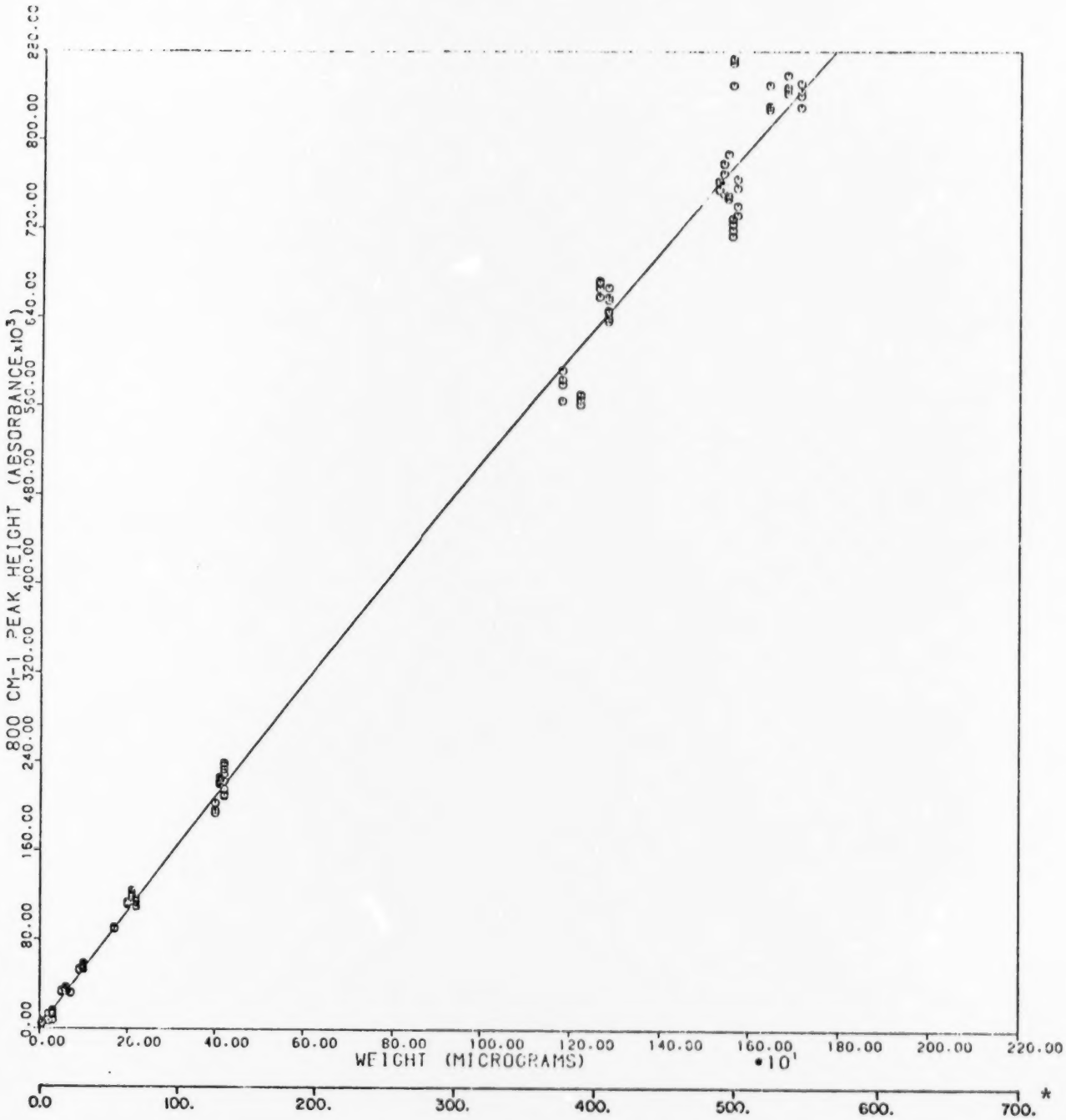


Figure 2. AIR DEPOSITED QUARTZ CALIBRATION CURVE

Plot of the 800 cm^{-1} peak height values and the calculated parabolic regression line.

*surface density of the quartz deposit ($\mu g/cm^2$)

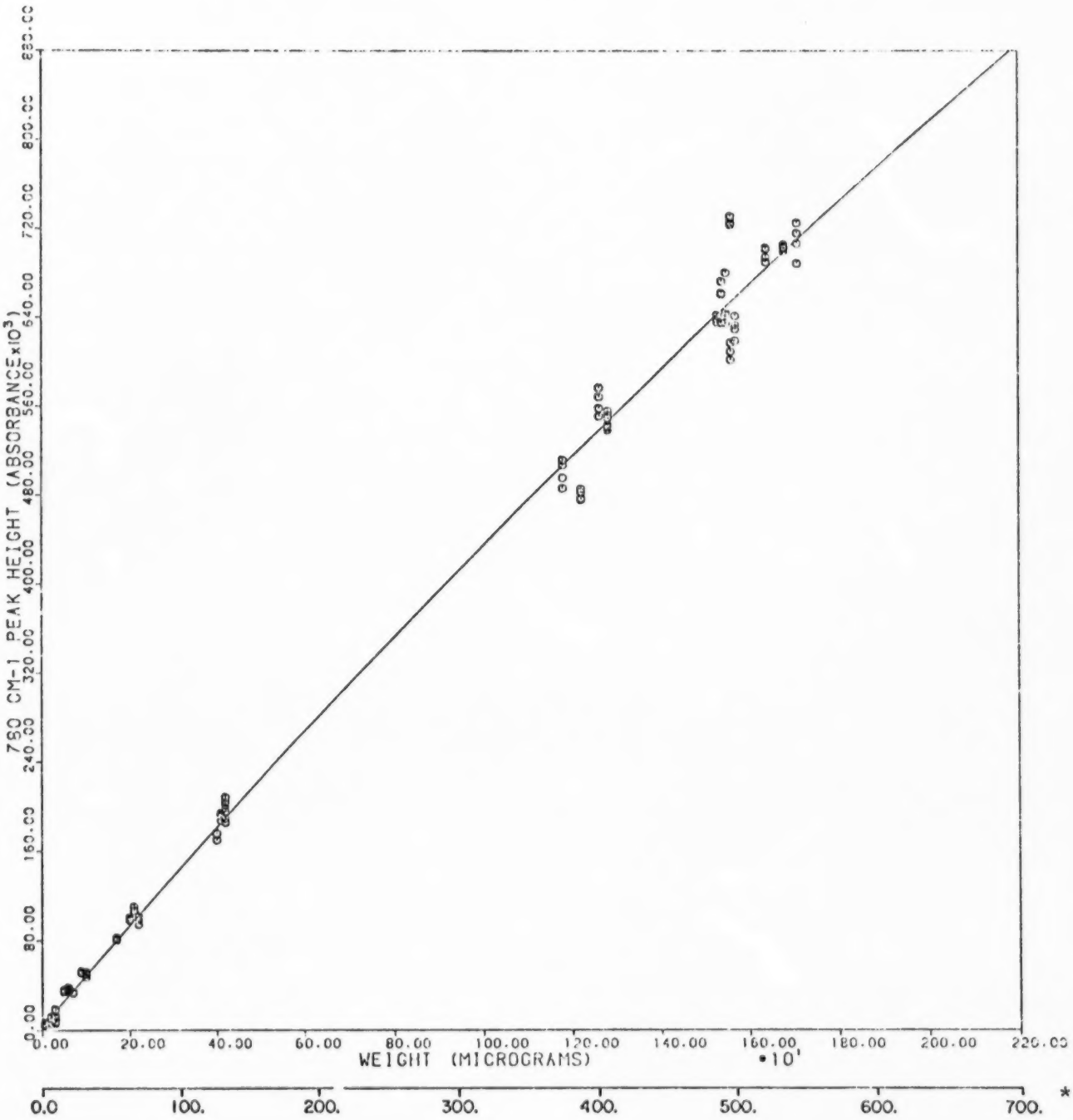


Figure 3. AIR DEPOSITED QUARTZ CALIBRATION CURVE

Plot of the 780 cm^{-1} peak height values and the calculated parabolic regression line.

*surface density of the quartz deposit ($\mu g/cm^2$)

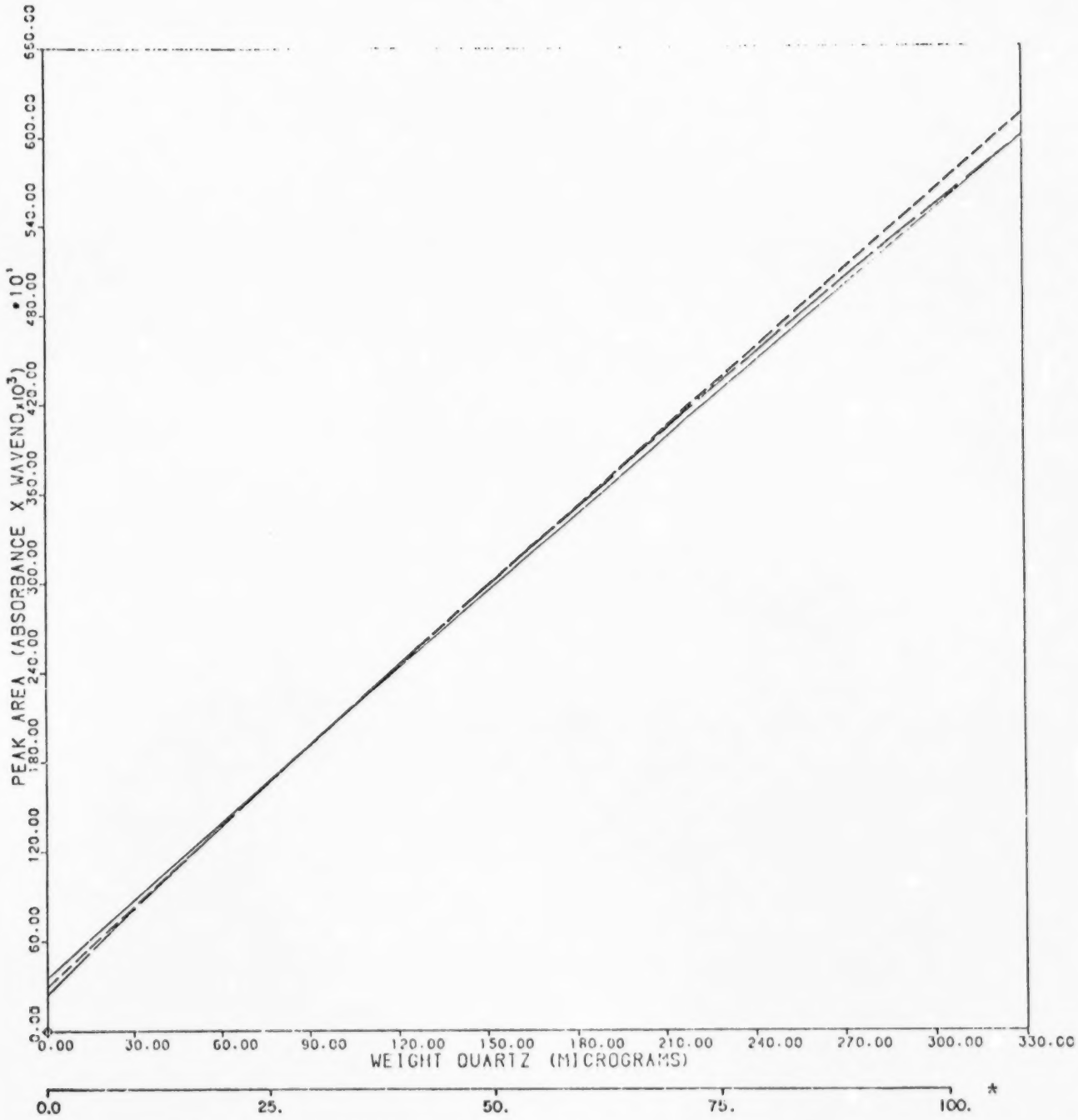


Figure 4. COMPARISON OF CALCULATED REGRESSIONS
Parabolic regression lines from equations in Table 1, 4 and 5 determined using standard sample weights:
≤1710 µg quartz ——————
≤420 µg quartz - - - - - - - - -
≤220 µg quartz — — — — — —

*surface density of the quartz deposit (µg/cm^2)

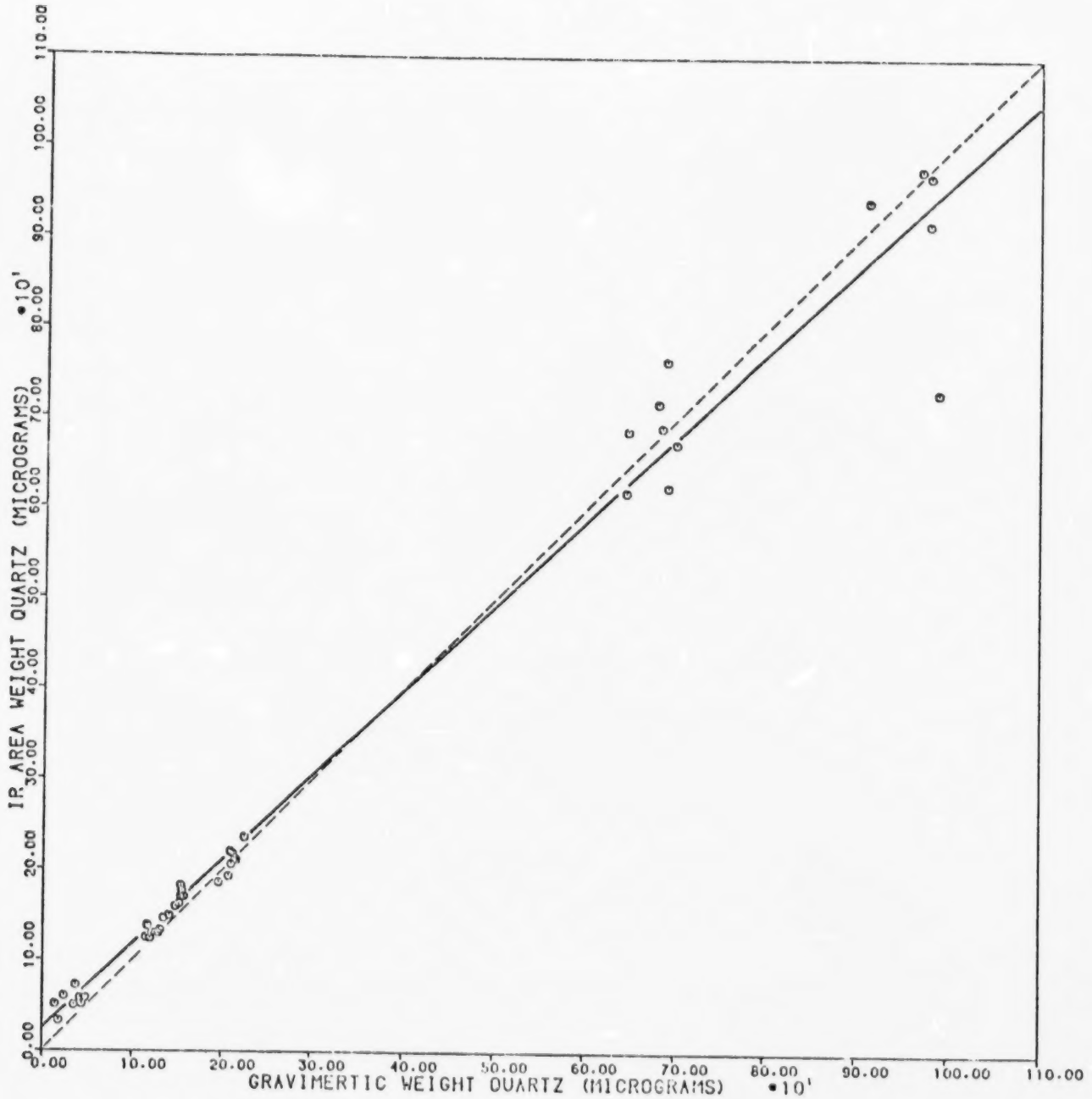


Figure 5. COMPARISON OF GRAV WT AND IR AREA WT

_ _ _ _ _ _ _ x = y

_____ linear regression line

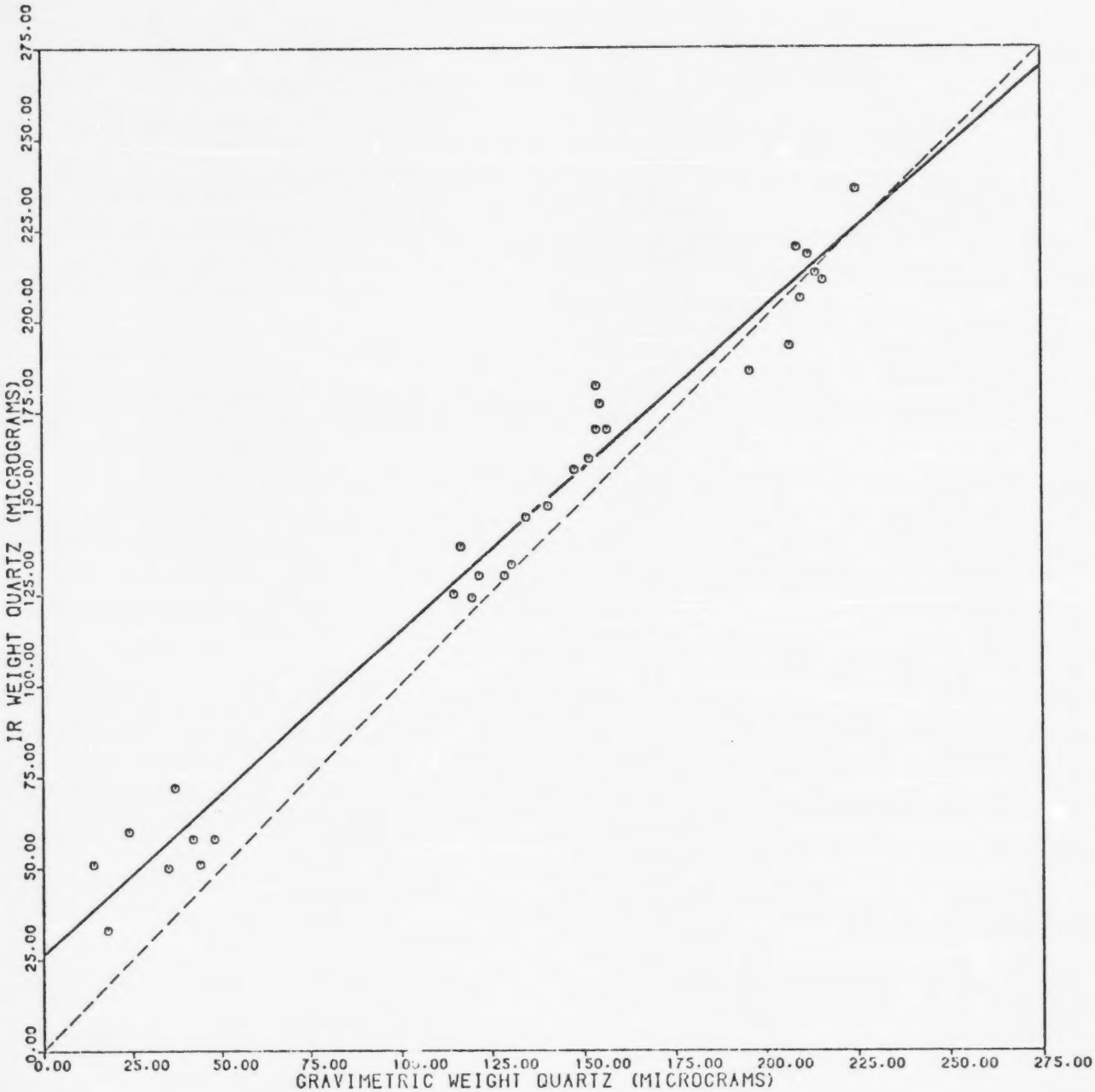


Figure 6. COMPARISON OF GRAV WT AND IR AREA WT
(expanded scale for plots in Figure 5)

_ _ _ _ _ _ _ x = y
_____ linear regression line

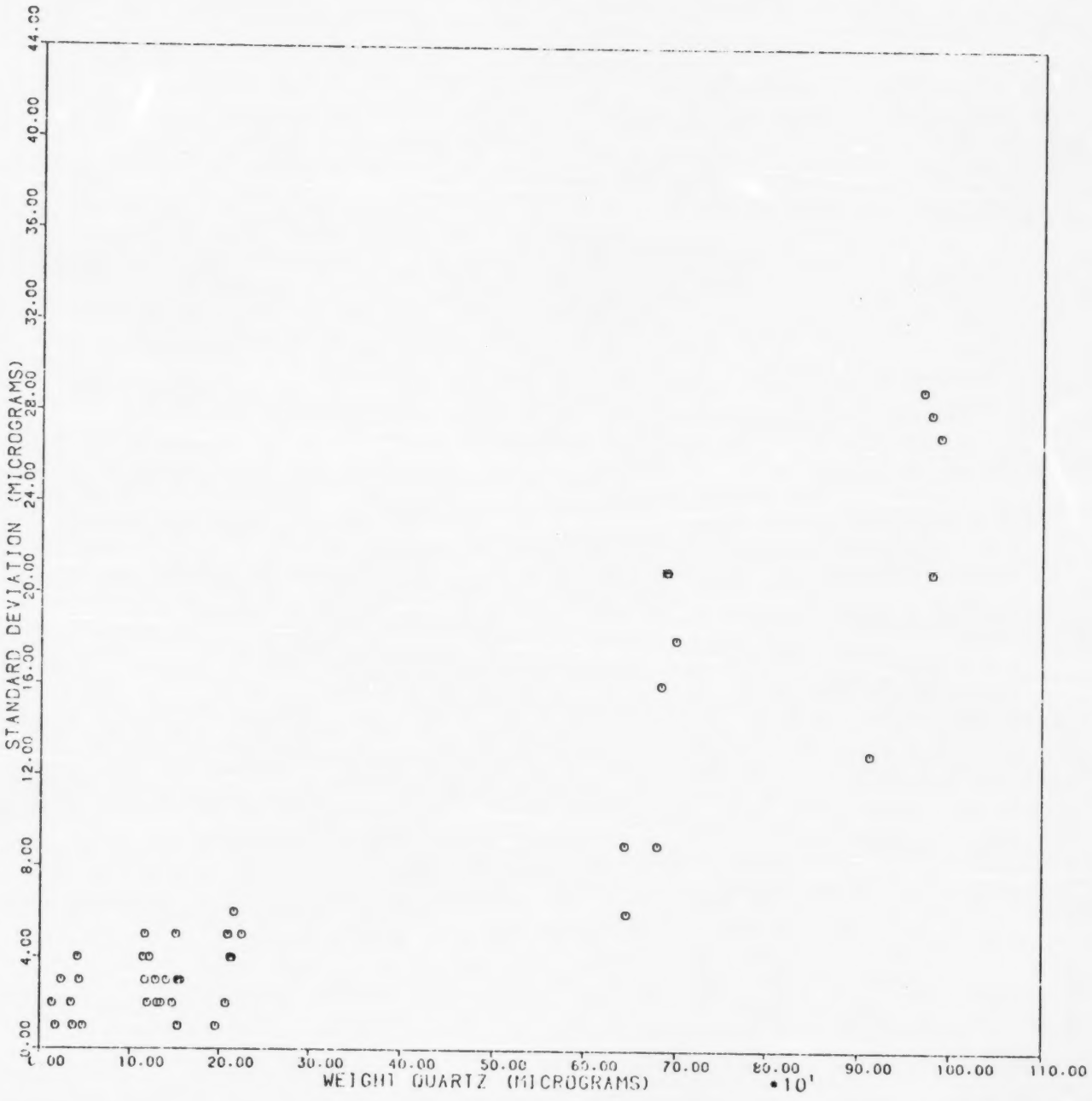


Figure 7. Standard deviation for IR determination plotted against weight of quartz.

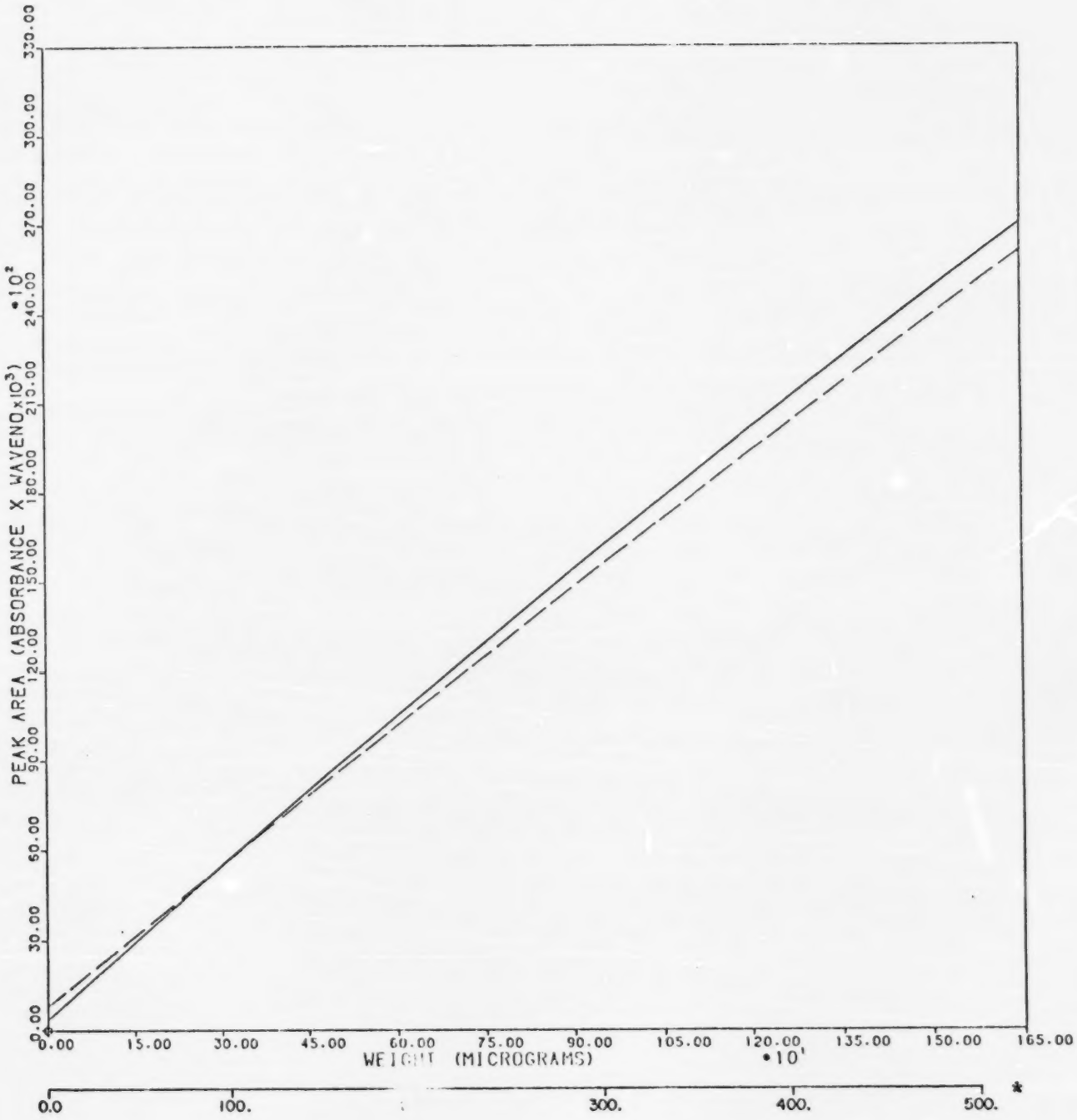


Figure 8. AIR DEPOSIT QUARTZ CALIBRATION CURVES

_ _ _ _ parabolic regression line calculated for 1978 air deposited Elliot Lake samples

_____ parabolic regression line from Table 1

*surface density of the quartz deposit (μg/cm²)

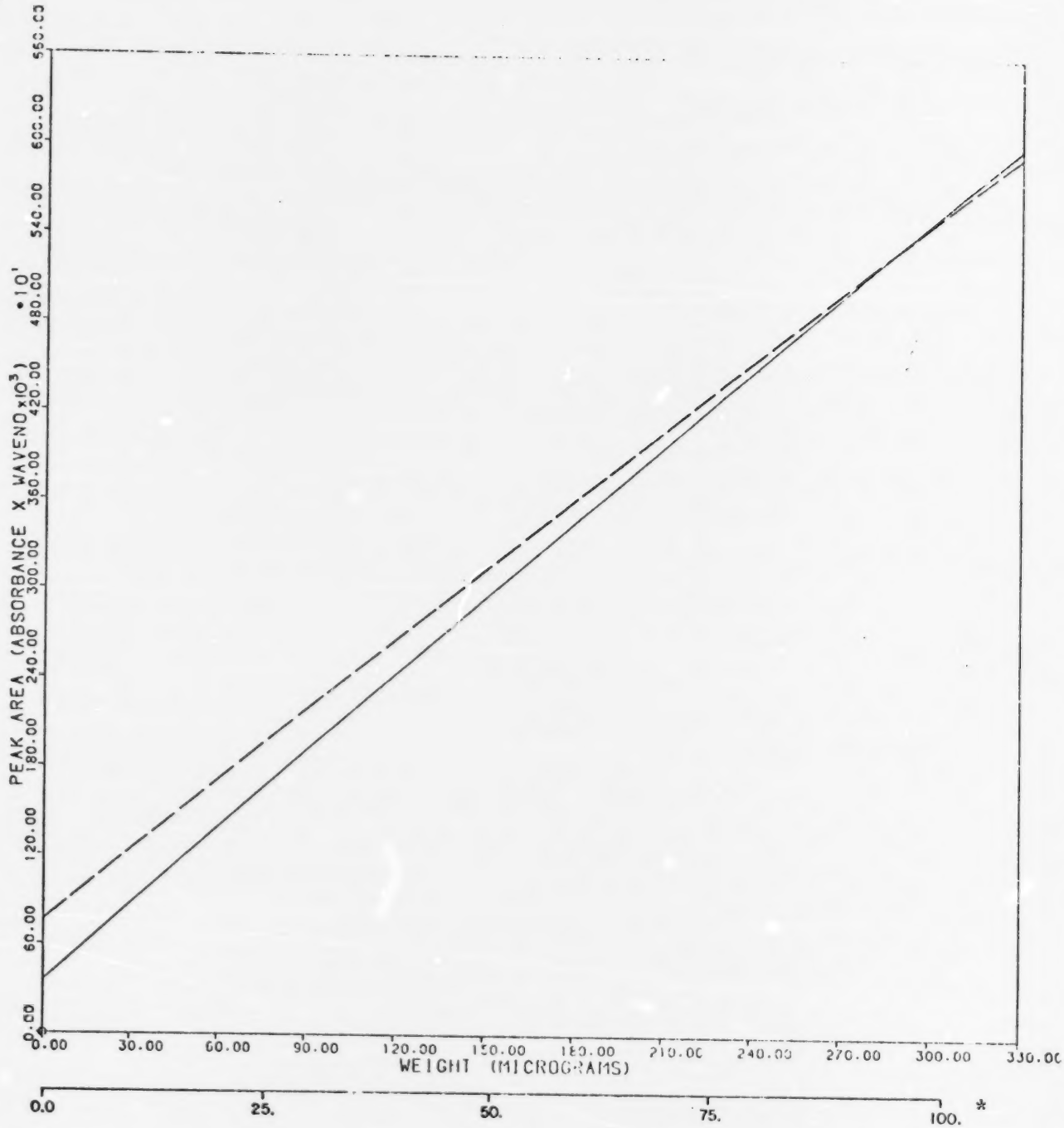


Figure 9. AIR DEPOSITED QUARTZ CALIBRATION CURVES

(expanded scale for plots in Figure 8)

_ _ _ _ _ _ _ _ parabolic regression line calculated for 1978 air deposited Elliot Lake samples

_____ parabolic regression line from Table 1

*surface density of the quartz deposit ($\mu g/cm^2$)

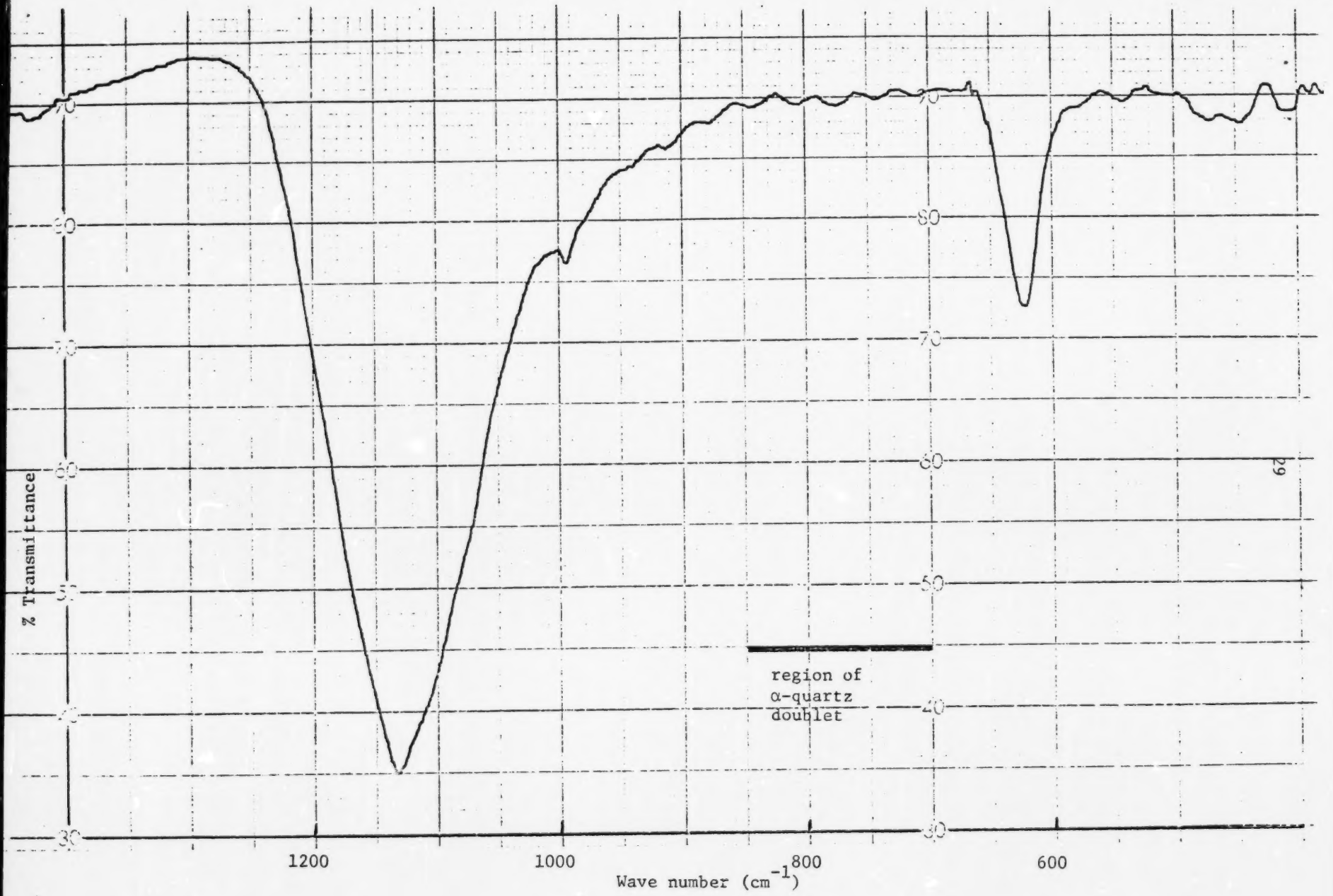


Figure 10. Residue of DM filter ashed at < 100°C

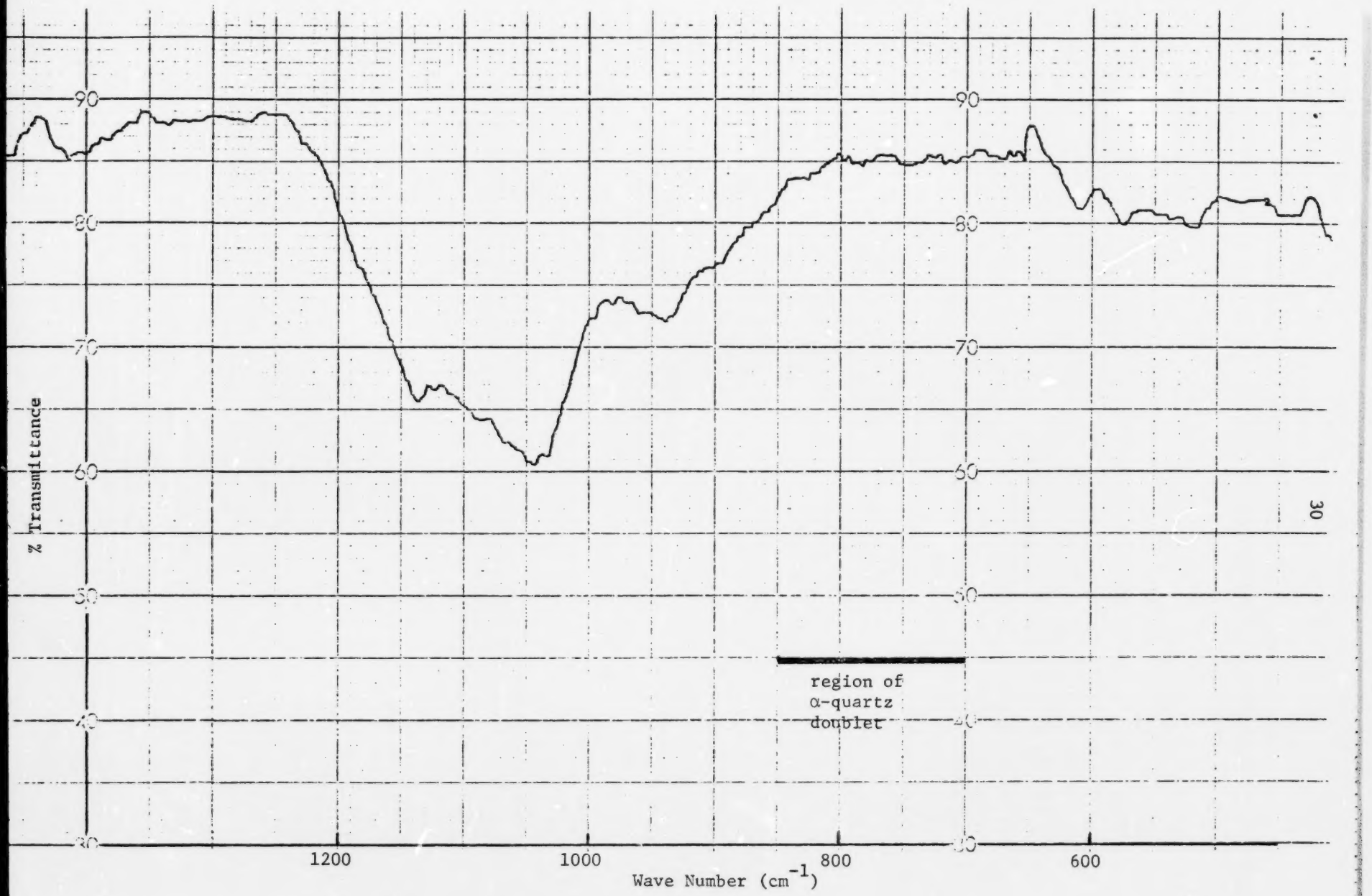
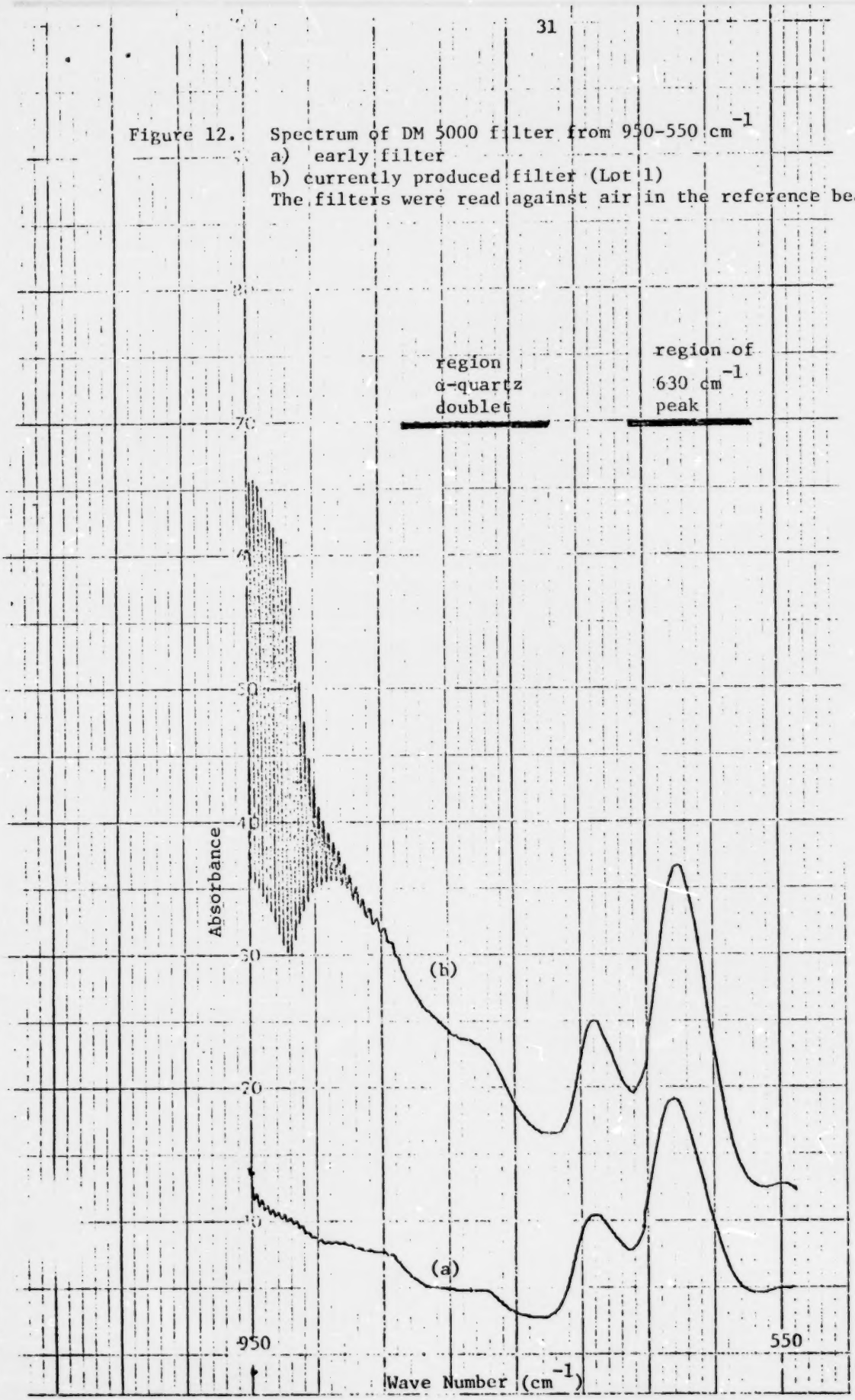


Figure 11. Residue of DM filter ashed at 650°C.

Figure 12. Spectrum of DM 5000 filter from 950-550 cm^{-1}
a) early filter
b) currently produced filter (Lot 1)
The filters were read against air in the reference beam.



ONEWAY - WTQUARTZ * (VARIABLE 2). CASES DIVIDED INTO GROUPS BASED ON VALUES OF * BOXNO * (VARIABLE 1)

```
                BOX 1                                   BOX 2

        ....................................+.....................................+.....
MIDPOINTS
 135.000)
 132.000)
 129.000)**
 126.000)*                                  *
 123.000)**
 120.000)M**
 117.000)                                   *
 114.000)                                   M****
 111.000)*
 108.000)*
 105.000)                                   ***
 102.000)
  99.000)
GROUP MEANS ARE DENOTED BY M"S IF THEY COINCIDE WITH *"S, N"S OTHERWISE

MEAN        120.740                         112.900
STD.DEV.      6.790                           6.340
S. E. M.      2.147                           2.005
MAXIMUM     128.700                         126.700
MINIMUM     106.700                         105.400
SAMPLE SIZE      10                              10

      ALL GROUPS COMBINED          ********************************* ANALYSIS OF VARIANCE TABLE *********************************
(EXCEPT CASES WITH UNUSED VALUES   *
  FOR BOXNO    )                   *   SOURCE              SUM OF SQUARES      DF      MEAN SQUARE    F VALUE    TAIL PROBABILITY
                                   *
MEAN            116.820            *   BETWEEN GROUPS          307.3280         1        307.3280        7.12          .0157
STD.DEV.          7.553            *   WITHIN  GROUPS          776.6640        18         43.1480
S. E. M.          1.689            *
MAXIMUM         128.700            *   TOTAL                  1083.9920        19
MINIMUM         105.400            ************************************************************************************************
SAMPLE SIZE          20
```

Figure 13. Comparison of weight of quartz obtained using filters from boxes from the same lot in the reference beam.

```
HISTOGRAM OF * WTQUARTZ * (VARIABLE     2). CASES DIVIDED INTO GROUPS BASED ON VALUES OF * LOTNO     * (VARIABLE     1)
          ************                                                 ************

        LOT 1                   LOT 2                   LOT 3                   LOT 4

        .....................+.....................+.....................+.....................+.....................
MIDPOINTS
 147.000)
 144.000)
 141.000)                    *
 138.000)
 135.000)                    *
 132.000)
 129.000)**                  *
 126.000)**                  M
 123.000)**                  *                       *
 120.000)***                 ***
 117.000)M                   **                      *
 114.000)*****                                       M*                      **
 111.000)*                                           ****                    M****
 108.000)*                                           *                       *
 105.000)***                                         *                       **
 102.000)
GROUP MEANS ARE DENOTED BY M'S IF THEY COINCIDE WITH *'S, M'S OTHERWISE

MEAN       116.820                 125.050                 112.740                 110.400
STD.DEV.     7.553                   7.977                   4.580                   2.954
S. E. M.     1.689                   2.522                   1.448                    .934
MAXIMUM    128.700                 140.900                 122.700                 114.600
MINIMUM    105.400                 118.100                 105.700                 105.700
SAMPLE SIZE     20                      10                      10                      10

      ALL GROUPS COMBINED           ********************************* ANALYSIS OF VARIANCE TABLE *********************************
(EXCEPT CASES WITH UNUSED VALUES    *
  FOR LOTNO    )                    *    SOURCE              SUM OF SQUARES      DF      MEAN SQUARE    F VALUE    TAIL PROBABILITY
                                    *
MEAN          116.366               *    BETWEEN GROUPS        1245.6512          3       415.2171        9.93          .0000
STD.DEV.        8.043               *    WITHIN  GROUPS        1923.9610         46        41.8252
S. E. M.        1.137               *
MAXIMUM       140.900               *    TOTAL                 3169.6122         49
MINIMUM       105.400               ********************************************************************************************
SAMPLE SIZE        50
```

Figure 14. Comparison of weight of quartz obtained using filters from different lots in the reference beam.

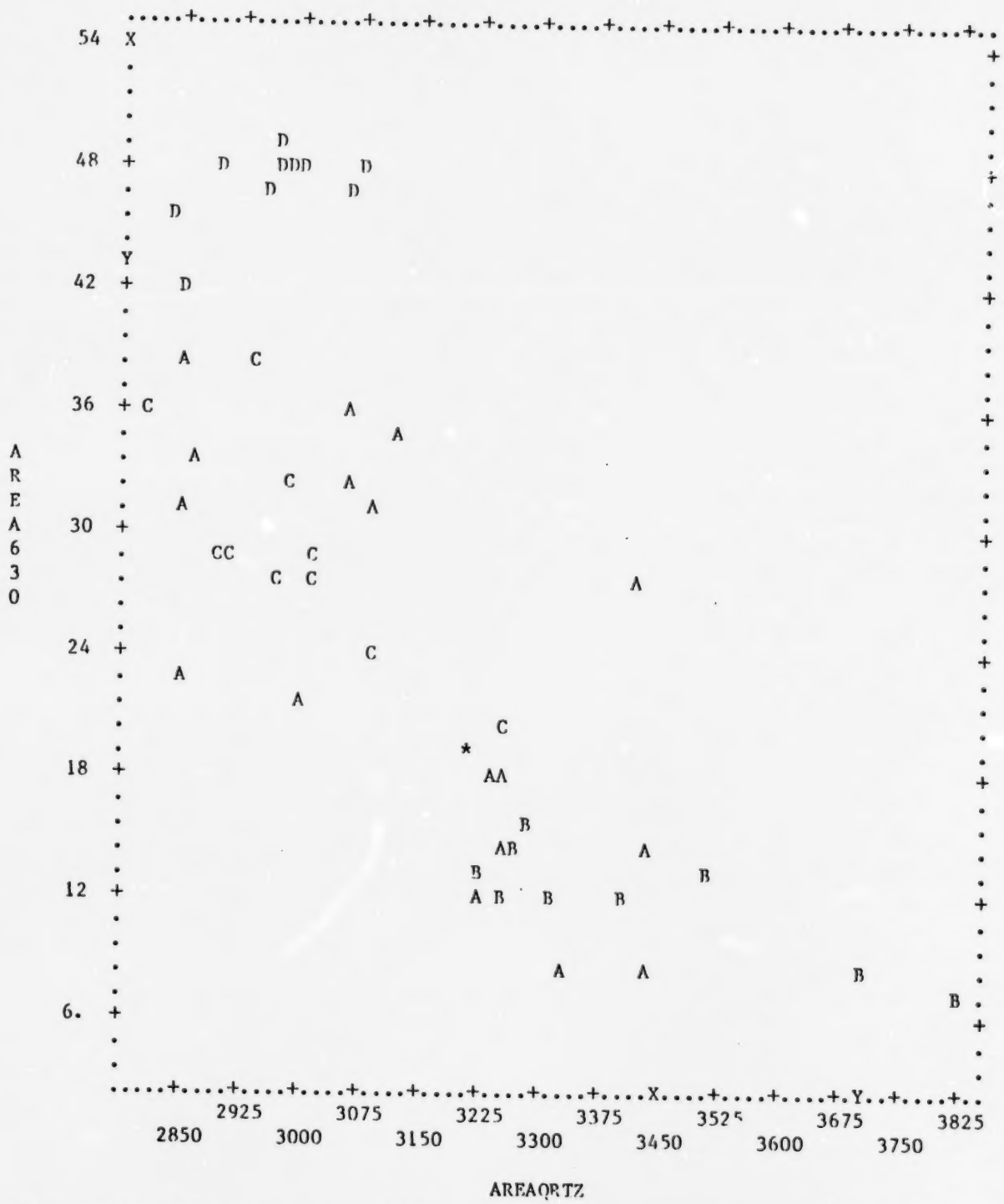


Figure 15. Plot of the area of the quartz doublet (absorbance x 10^3 x cm^{-1}) vs area of the 630 cm^{-1} band (absorbance x 10^3 x cm^{-1}) of the DM filter when filters from Lot 1 (A), Lot 2 (B), Lot 3 (C) and Lot 4 (D) are used in the reference beam.

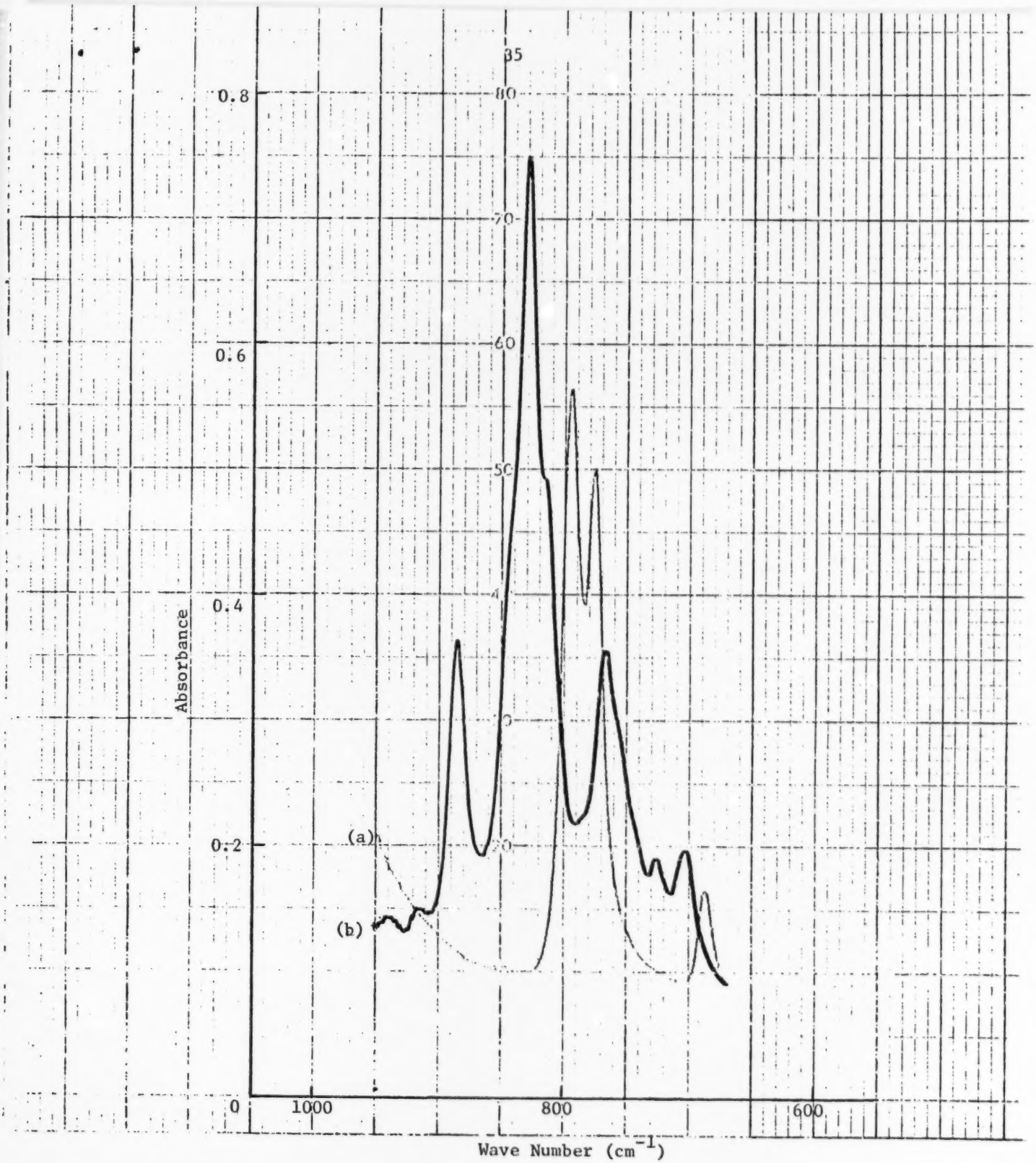


Figure 16. Permanent standards
a) α quartz doublet
b) carbonate peak (Nuclepore filter)

APPENDIX

Air deposited quartz calibration curve data

No.	Gravimetric weight (μg)	Peak area (absorbance x cm^{-1})		800 cm^{-1} peak height (absorbance)		780 cm^{-1} peak height (absorbance)	
		Mean †	Stand. † dev.	Mean †	Stand. † dev.	Mean †	Stand. † dev.
1	1260	21.924	.379	.667	.006	.536	.011
2	1180	19.411	.372	.580	.012	.500	.011
3	1280	21.531	.349	.657	.009	.550	.005
4	1280	20.968 †	.010 †	.640 †	.004 †	.540 †	.001 †
5	1220	18.681	.238	.567	.004	.480	.005
6	1560	28.544	.339	.866	.011	.726	.003
7	1540	25.614	.513	.768	.012	.654	.016
8	1530	25.042	.092	.759	.004	.639	.003
9	1570	24.657	.462	.749	.015	.630	.009
10	1560	23.812	.252	.721	.007	.609	.006
11	420	8.162	.171	.234	.004	.204	.004
12	400	70.36	.146	.199	.005	.173	.003
13	410	7.725	.077	.223	.002	.192	.003
14	410	7.783	.038	.221	.001	.193	.001
15	420	7.617	.212	.214	.006	.189	.004
16	210	4.318	.076	.121	.002	.108	.002
17	170	3.325	.041	.090	.001	.081	.001
18	200	3.988	.029	.112	.001	.099	.001
19	220	3.964	.078	.111	.002	.096	.002
20	220	4.019	.083	.115	.002	.100	.002
21	100	2.104	.025	.055	.001	.050	.0004
22	100	1.974	.043	.053	.0003	.048	.0000
23	100	2.149	.039	.057	.001	.051	.001
24	100	2.150	.006	.058	.0004	.052	.0004
25	90	2.067	.035	.053	.0005	.052	.001

Appendix (Cont'd)

No.	Gravimetric weight (µg)	Peak area (absorbance x cm^{-1})		800 cm^{-1} peak height (absorbance)		780 cm^{-1} peak height (absorbance)	
		Mean †	Stand. † dev.	Mean †	Stand. † dev.	Mean †	Stand. † dev.
26	60	1.482	.074	.035	.0001	.037	.001
27	70	1.333	.040	.032	.001	.034	.0005
28	60	1.609	.022	.036	.0004	.038	.0004
29	60	1.450	.007	.033	.0004	.035	.0005
30	50	1.406	.028	.033	.001	.035	.001
33	30	.846	.021	.016	.0004	.018	.0004
34	30	.801	.040	.015	.001	.018	.001
35	30	.542	.008	.014	.0003	.012	.0002
36	30	.691	.070	.013	.0001	.012	.0002
38	20	.734	.064	.013	.0002	.011	.0004
39	30	.627	.104	.012	.001	.012	.001
40	30	.616	.031	.013	.003	.012	.0004
42	30	.493	.097	.007	.0000	.006	.0005
43	30	.691	.167	.008	.0005	.008	.001
44	20	.639	.090	.007	.0005	.011	.001
45	20	.636	.122	.007	.001	.006	.001
46	7	.698 †	.144 †	.007 †	.001 †	.006 †	.0001 †
47	5	.422	.008	.004	.0003	.005	.0002
50	8	.546	.063	.006	.0001	.006	.0004
51	1710	27.891	.511	.841	.009	.709	.016
52	1550	25.470	.760	.759	.019	.650	.020
53	1640	27.498	.161	.835	.010	.697	.006
54	1680	27.659	0.70	.849	.007	.703	.002
55	1660	27.027	.253	.796	.012	.681	.007

† values calculated on four readings except for sample numbers 4 and 4b.

† values calculated on five readings.